

PATENT ABSTRACTS OF JAPAN

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(54) PURIFICATION OF MODIFIED SILICONE OIL

(57)Abstract:

PURPOSE: To efficiently purify a modified silicone oil in highly safe and economical way by bringing a specific modified silicone oil into contact with carbon dioxide being in a e.g. subcritical state to extract and separate the low- molecular weight matter in the oil.

CONSTITUTION: A modified silicone oil consisting of a polysiloxane bearing in the chain functional group(s) selected from epoxy group, carboxyl group, hydroxyl group, amino group, (substituted) vinyl group and -O-(CH₂-CH(R')-O-)n-H (n is 1-25; R' is H or CH₃) is brought into contact with carbon dioxide being in subcritical or supercritical state to extract and separate the low-molecular weight matter in the silicone oil, thus accomplishing the objective purification.

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CLAIMS

[Claim(s)]

[Claim 1] In a chain, an epoxy group, a carboxylic-acid radical, a hydroxyl group, the amino group, the vinyl group which is not permuted [a permutation or] -- and -- -O-(CH₂-CH(R'))-O- n-H (n = 1-25 and R' express H or CH₃) from -- the denaturation silicone oil which consists of a polysiloxane which has the functional group chosen The purification approach of denaturation silicone oil which consists of making the carbon dioxide of subcritical or a supercritical condition contact, and carrying out extraction separation of the low-molecular-weight matter in this silicone oil.

[Claim 2] Denaturation silicone oil is Formula I.;

--Si(CH₃)₂-O-- The dimethylsiloxane unit expressed with (I), and formula II;

--Si(R) (CH₃)-O-- (II) (the inside of a formula and R are [Formula 1])

-- O - C H₂ - C H - C H₂ .



- O-(CH (R')) n-COOH (n = 1-20) and -O-(CH (R')) n-OH (n = 1-20), - O-(CH₂-CH (R')) n-OH (n = 1-25), -O-(CH (R')) n-NH₂ (n = 1-20) - O-CH=CH₂ and -O-CH₂-C(R') =CH₂ -O-(CH (R')) n-O-CO-C (R') =CH₂ -- and (n = 1-10) -- Among a -O-(CH₂-CH(R')-O-) n-H [(n = 1-25) type R' and R'' are each. H or CH₃ It is chosen from] to express. The approach according to claim 1 of consisting of a copolymer polysiloxane which consists of functional-group content siloxane units expressed.

[Claim 3] Denaturation silicone oil is the following formula.;

CH₂=C(R')-COO-(Si(CH₃)₂-O-) n - CO-C = CH₂, CH₂=C (R'') (R')-CO-O-CH₂-CH(OH)-CH₂-O-(Si (CH₃)₂-O-) n -- CH₂-CH(OH)-CH₂-O-CO-C(R'') =CH₂ and CH₂=C(R')-CH₂-O-(Si(CH₃)₂-O-) n -

The approach according to claim 1 of consisting of terminal modification poly dimethylsiloxane expressed by either of CH₂-C(R'') =CH(s)2 (n = 1-300, R', and R'' express H or CH₃ among a formula, respectively).

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the silicone oil (henceforth "denaturation silicone oil") purification approach which consists of a polysiloxane containing a functional group, and relates to the purification approach of this denaturation silicone oil which becomes a detail from carrying out extraction separation of the low-molecular-weight matter contained in it from denaturation silicone oil by extract with the carbon dioxide of subcritical or a supercritical condition more.

[0002]

[A technical background] Although general silicone oil consists of a homopolymer of dimethylsiloxane, the denaturation silicone oil containing functional groups, such as a glycidyl group, a carboxylic-acid radical, an amino group, and a hydroxyl group, or polyethylene-glycol denaturation, the silicone oil that carried out polypropylene-glycol denaturation are used for the application which needs an improvement of the spreading engine performance etc.

[0003] The low-molecular-weight matter which is mainly an unreacted object and oligomer is included in these silicone oil after manufacture by the polymerization of a siloxane, and since degradation is caused, it is necessary to remove these. Although these are removed from the former by usual vacuum distillation or a usual molecular distillation method, in order to attain clearance of the desired low-molecular-weight matter, very severe conditions must be maintained strictly for a long time, and, moreover, it is necessary to repeat such vacuum distillation or molecular distillation usually two or more times. Therefore, the approach the actual condition is that purification of the denaturation silicone oil by vacuum distillation or the molecular distillation method has also required costs with technical difficulty, and it can remove efficiently the low-molecular-weight matter in these conversion silicone oil cheaply is searched for.

[0004] On the other hand, the supercritical extraction which used the carbon dioxide (carbon dioxide gas) is known from Men, such as a fluid of a supercritical condition especially safety, and profitability, as the extract approach of a minute amount organic compound, and it is used for purification of the matter or the extract of the useful matter. About the application to purification of the silicone oil of this supercritical extraction, the low-molecular-weight matter in the silicone oil which consists of a dimethylsiloxane homopolymer dissolved in the carbon dioxide of a supercritical condition, and although it was known that it can refine, the knowledge about the denaturation silicone oil containing the siloxane containing a functional group was not reported at all.

[0005]

[The technical problem which should solve invention] Therefore, the object of this invention is offering the approach supercritical extraction's being able to extract the low-molecular-weight matter from the denaturation silicone oil which consists of a polysiloxane containing a functional group efficiently, and denaturation silicone oil's being refined.

[0006]

[Means for Solving the Problem] In order to attain the object of said this invention, as a result of

examining the solubility to the carbon dioxide of the supercritical condition of the low-molecular-weight matter in the silicone oil containing the siloxane containing various kinds of functional groups, when it was the denaturation silicone oil containing the siloxane which has a fixed functional group, it became clear by the supercritical extraction in a carbon dioxide for desired purification to be possible.

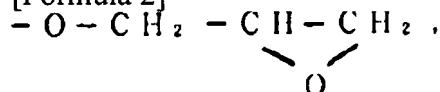
[0007] In a chain this invention Therefore, an epoxy group, a carboxylic-acid radical, a hydroxyl group, amino group, the vinyl group which is not permuted [a permutation or] -- and -- -O-(CH₂-CH(R')-O-) n-H (n = 1-25 and R' express H or CH₃) from -- the silicone oil which consists of a polysiloxane which has the functional group chosen It is the purification approach of silicone oil which consists of making the carbon dioxide of subcritical or a supercritical condition contact, and carrying out extraction separation of the low-molecular-weight matter in this silicone oil.

[0008] As a desirable example of the denaturation silicone oil which can be refined, it is Formula I by the approach of this invention.;

--Si(CH₃)₂-O-- The dimethylsiloxane unit expressed with (I), and formula II;

--Si(R) (CH₃)-O-- (II) (the inside of a formula and R are [0009])

[Formula 2]



[0010] - O-(CH (R')) n-COOH (n = 1-20) and -O-(CH (R')) n-OH (n = 1-20), - O-(CH₂-CH (R')) n-OH (n = 1-25), -O-(CH (R')) n-NH₂ (n = 1-20) - O-CH=CH₂ and -O-CH₂-C(R')=CH₂ -O-(CH (R')) n-O-CO-C(R')=CH₂ -- and (n = 1-10) -- Among a -O-(CH₂-CH(R')-O-) n-H [(n = 1-25) type R' and R" are each. H or CH₃ It expresses. the time of two or more R' and/or R" existing in one radical -- respectively -- H or CH₃ from -- it is chosen from] chosen independently The denaturation silicone oil which consists of a copolymer polysiloxane which consists of functional-group content siloxane units expressed is mentioned.

[0011] Moreover, the approach of this invention is applicable also to the denaturation silicone oil which becomes a siloxane chain end from the polysiloxane which has the above functional groups. As an example of such a terminal modification polysiloxane, it is the following type.;

CH₂=C(R')-COO-(Si(CH₃)₂-O-) n-CO-C(R'')=CH₂ (III) CH₂=C(R')-CO-O-CH₂-CH(OH)-CH₂-O-(Si(CH₃)₂-O-) n- -CH₂-CH(OH)-CH₂-O-CO-C(R'')=CH₂, (IV) CH₂=C(R')-CH₂-O-(Si(CH₃)₂-O-) n-CH₂-C(R'')=CH₂ (V) (n = 1-300, R', and R" express H or CH₃ among a formula, respectively) The terminal modification poly dimethylsiloxane expressed by either is mentioned.

[0012] Such terminal modification poly dimethylsiloxane denaturalizes by the vinyl monomer which corresponds poly dimethylsiloxane, and is manufactured, and, usually the unreacted vinyl monomer after manufacture is included. Since such an unreacted vinyl monomer was exposed to the elevated temperature when it tended to remove by conventional vacuum distillation and molecular distillation, it occurred thermal polymerization, and it had the inconvenience that clearance became difficult. On the other hand, by this invention approach, since it is not exposed to such an elevated temperature, the thermal polymerization of the vinyl monomer is not carried out, but moreover, it may dissolve in the carbon dioxide of supercritical or a subcritical state, and a vinyl monomer may fully be removed.

[0013] The low-molecular-weight matter removed by this invention approach means the oligomer which consists of units, such as an unreacted object of a functional-group content siloxane and dimethylsiloxane, and it to about ten pieces. Moreover, especially the molecular weight of the polysiloxane which constitutes the silicone oil set as the object of purification by this invention approach is usually abbreviation, although not limited. It is preferably applicable about the silicone oil which consists of a polysiloxane including the siloxane unit to about 400.

[0014] The critical points of a carbon dioxide are about 31.0 degrees C and about 75.3kg/cm², and it is referred to as being in a supercritical condition to be in the condition of having exceeded this and to have the in-between property of a liquid and a gas, i.e., the consistency near a liquid, and a diffusion coefficient near a gas. Moreover, although there is no clear definition, generally a subcritical state exists

near the supercritical condition, is about 50kg/cm² in the temperature of about 25 degrees C or more, and pressure in a carbon dioxide, is in the above-mentioned critical temperature or below a pressure, and says what has the in-between property of the above liquids and a gas.

[0015] When the conditions used for the usual supercritical extraction and a subcritical extract can be used in operation of this invention approach and 80 - 500 kg/cm², 50 to 120 degree C, and a subcritical extract are usually included in the case of supercritical extraction, it is desirable to carry out using the carbon dioxide in a supercritical condition or a subcritical state under 70 - 500 kg/cm² and conditions of about 25-120 degrees C. The equipment used for the usual supercritical extraction and a subcritical extract can be used for the equipment for enforcing this invention approach. this invention approach can be performed to drawing 1 using the equipment shown roughly. In drawing 1 , a carbon dioxide is supplied to a compressor 3 via a heat exchanger 2 from the carbon-dioxide cylinder 1, and it compresses to a predetermined pressure. The carbon dioxide compressed into the predetermined pressure is further made into extract temperature predetermined by the heat exchanger 4, and it is made supercritical or a subcritical state, and introduces to the extract tub 5. The extract tub 5 prepared by predetermined temperature is filled up with the conversion silicone oil which should be refined, the carbon dioxide containing the low-molecular-weight matter after extracting is decompressed through a reducing valve 6, it introduces into the separation tub 7, and the low-molecular-weight matter is separated. The carbon dioxide which separated the low-molecular-weight matter is recycled via a heat exchanger 2.

[0016]

[Effect of the Invention] The low-molecular-weight matter is efficiently extracted from the denaturation silicone oil which consists of a polysiloxane containing a functional group by this invention, and the advantageous method of refining denaturation silicone oil easily and cheaply is offered. Moreover, according to this invention approach, even if it is the denaturation silicone oil containing a vinyl monomer, it can remove, without carrying out the thermal polymerization of the vinyl monomer.

[0017]

[Example] Hereafter, an example explains this invention further. The rate of the functional-group content siloxane number of unit to the total siloxane number of unit in the denaturation silicone oil concerned shows the functional-group content of denaturation silicone oil among an example. Example 1 bore of 20mm, container made from a stainless steel with a die length of 40cm (It is thickness to an upper bed and a soffit. 2mm porous sheet made from titanium) (10 micrometers of diameters of pore) -- wearing from -- epoxy denaturation silicone oil which becomes the becoming extract tub from the copolymer of the dimethylsiloxane / 3-glycidoxy propyl oxymethyl siloxane of 30.1 g (about 8000 viscosity cps (25 degrees C) and average molecular weight 17500 --) About 30% of epoxy group content, low-molecular-weight matter content abbreviation 9.6 % of the weight It is filled up and is the carbon dioxide of the supercritical condition of 80 degrees C and 350 kg/cm². It supplied upwards for about 15 minutes from the container soffit at the rate of 6 l/min, about 2.9g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0018] the silicone oil obtained after extract purification -- GPC (Gel Permeation Chromatography) when it analyzes, it shows that the low-molecular-weight matter is not substantially contained in this silicone oil -- having -- moreover, the viscosity -- 8000 cps before processing (25 degrees C) from -- 8960 cps (25 degrees C) It was going up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be carried out the product made from example dispermy, about 2500 viscosity cps (25 degrees C) Carboxylic-acid denaturation silicone oil 29.5 g which consists of dimethylsiloxane / a carboxy methoxymethyl siloxane of a mean molecular weight 6500, about 12% of carboxylic-acid radical content, and about 10.5 % of the weight of low-molecular-weight matter content is used. Except having supplied the carbon dioxide of 75 degrees C and the supercritical condition of 310 kg/cm² for about 10 minutes by rate-of-flow 6 l/min, silicone oil was refined by the same approach as an example 1, about 3.1g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0019] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by

the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 2500 cps before processing (25 **) from -- 2980 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 3, about 1500 viscosity cps (25 degrees C) Average molecular weight 4000, about 15% of hydroxy group content, low-molecular-weight matter content abbreviation Alcoholic conversion silicone oil 29.8 g which consists of 8.4% of the weight of dimethylsiloxane / a hydroxy ethoxymethyl siloxane is used. Except having supplied the carbon dioxide of 75 degrees C and the supercritical condition of 290 kg/cm² for about 10 minutes by rate-of-flow 6 l/min, silicone oil was refined by the same approach as an example 1, about 2.5g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0020] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 1500 cps before processing (25 **) from -- 1650 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 4, about 1300 viscosity cps (25 degrees C) A mean molecular weight 4800, low-molecular-weight matter content abbreviation 7.9% of the weight of dimethylsiloxane unit -(Si(CH₃)(-O-(CH₂CH₂O)4H)-O)- It consists of a unit. (mole ratio 3:1)

Tetraethylene glycol denaturation silicone oil 30.5 g is used. It is the rate of flow about the carbon dioxide of 80 degrees C and the supercritical condition of 400 kg/cm². Except having supplied for about 11 minutes by 7 l/min, silicone oil was refined by the same approach as an example 1, about 2.4g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0021] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 1300 cps before processing (25 **) from -- 1430 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 5, about 3000 viscosity cps (25 degrees C) Vinyl denaturation silicone oil 28.8g which consists of dimethylsiloxane / an aryloxymethyl siloxane of average molecular weight 7800, about 25% of allyl group content, and about 11.8 % of the weight of low-molecular-weight matter content is used. It is the rate of flow about the carbon dioxide of 50 degrees C and the supercritical condition of 240 kg/cm². Except having supplied for about 10 minutes by 6 l/min, silicone oil was refined by the same approach as an example 1, about 3.4g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0022] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 3000 cps before processing (25 **) from -- 3360 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 6, about 4000 viscosity cps (25 degrees C) Average molecular weight 10400, about 15% of methacrylate radical content, low-molecular-weight matter content abbreviation Methacrylate denaturation silicone oil 29.7 g which consists of 7.0% of the weight of dimethylsiloxane / a methacryloxypropyl oxymethyl siloxane is used. 50 degrees C, It is the rate of flow about the carbon dioxide of a 305kg/cm² supercritical condition. Except having supplied for about 10 minutes by 6 l/min, silicone oil was refined by the same approach as an example 1, about 2.1g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0023] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 4000 cps before processing (25 **) from -- 4410 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 7, about 1500 viscosity cps (25 degrees C) Average molecular weight 4300, amino-group content about 5 %, and about 12.0 % of the weight of low-molecular-weight matter content, Dimethylsiloxane unit -(Si(CH₃)(-O-(CHCH₃)₅-NH₂)-O)- Amino-group denaturation silicone oil 30.2 g which consists of a unit is used. It is the rate of flow about the

carbon dioxide of 80 degrees C and the supercritical condition of 350 kg/cm². Except having supplied for about 20 minutes by 6 l/min, silicone oil was refined by the same approach as an example 1, about 3.8g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub. [0024] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 1500 cps before processing (25 **) from -- 1760 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 8, about 1500 viscosity cps (25 degrees C) It is radical-CH₂-CCH₃=CH₂ (n is abbreviation at said formula V by 200) to both ends of a mean molecular weight 35000, vinyl group content about 0.6 %, and about 13.0 % of the weight of low-molecular-weight matter content. Poly dimethylsiloxane which it has R' and R" -- CH₃ Thing Becoming terminal modification silicone oil 29.0 g is used. from -- It is the rate of flow about the carbon dioxide of 70 degrees C and the supercritical condition of 250 kg/cm². Except having supplied for about 20 minutes by 6 l/min, silicone oil was refined by the same approach as an example 1, about 3.9g low-molecular-weight matter was extracted, and uptake was carried out to the separation tub.

[0025] it shows that the low-molecular-weight matter is not substantially contained in this silicone oil by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 1500 cps before processing (25 **) from -- 1800 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

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MEANS

[Means for Solving the Problem] In order to attain the object of said this invention, as a result of examining the solubility to the carbon dioxide of the supercritical condition of the low-molecular-weight matter in the silicone oil containing the siloxane containing various kinds of functional groups, when it was the denaturation silicone oil containing the siloxane which has a fixed functional group, it became clear by the supercritical extraction in a carbon dioxide for desired purification to be possible.

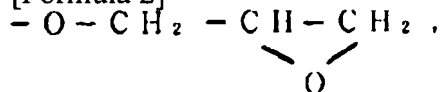
[0007] In a chain this invention Therefore, an epoxy group, a carboxylic-acid radical, a hydroxyl group, amino group, the vinyl group which is not permuted [a permutation or] -- and -- -O-(CH₂-CH(R'))-O-) n-H (n = 1-25 and R' express H or CH₃) from -- the silicone oil which consists of a polysiloxane which has the functional group chosen It is the purification approach of silicone oil which consists of making the carbon dioxide of subcritical or a supercritical condition contact, and carrying out extraction separation of the low-molecular-weight matter in this silicone oil.

[0008] As a desirable example of the denaturation silicone oil which can be refined, it is Formula I by the approach of this invention.;

--Si(CH₃)₂-O-- The dimethylsiloxane unit expressed with (I), and formula II;

--Si(R) (CH₃)-O-- (II) (the inside of a formula and R are [0009])

[Formula 2]



[0010] - O-(CH (R')) n-COOH (n = 1-20) and -O-(CH (R')) n-OH (n = 1-20), - O-(CH₂-CH (R')) n-OH (n = 1-25), -O-(CH (R')) n-NH₂ (n = 1-20) - O-CH=CH₂ and -O-CH₂-C(R')=CH₂ -O-(CH (R')) n-O-CO-C(R')=CH₂ -- and (n = 1-10) -- Among a -O-(CH₂-CH(R'))-O-) n-H [(n = 1-25) type R' and R" are each. H or CH₃ It expresses. the time of two or more R' and/or R" existing in one radical -- respectively -- H or CH₃ from -- it is chosen from] chosen independently The denaturation silicone oil which consists of a copolymer polysiloxane which consists of functional-group content siloxane units expressed is mentioned.

[0011] Moreover, the approach of this invention is applicable also to the denaturation silicone oil which becomes a siloxane chain end from the polysiloxane which has the above functional groups. As an example of such a terminal modification polysiloxane, it is the following type.;

CH₂=C(R')-COO-(Si(CH₃)₂-O-) n-CO-C(R'')=CH₂ (III) CH₂=C(R')-CO-O-CH₂-CH(OH)-CH₂-O-(Si(CH₃)₂-O-) n- -CH₂-CH(OH)-CH₂-O-CO-C(R'')=CH₂, (IV) CH₂=C(R')-CH₂-O-(Si(CH₃)₂-O-) n-CH₂-C(R'')=CH₂ (V) (n = 1-300, R', and R" express H or CH₃ among a formula, respectively) The terminal modification poly dimethylsiloxane expressed by either is mentioned.

[0012] Such terminal modification poly dimethylsiloxane denaturalizes by the vinyl monomer which corresponds poly dimethylsiloxane, and is manufactured, and, usually the unreacted vinyl monomer after manufacture is included. Since such an unreacted vinyl monomer was exposed to the elevated temperature when it tended to remove by conventional vacuum distillation and molecular distillation, it occurred thermal polymerization, and it had the inconvenience that clearance became difficult. On the

other hand, by this invention approach, since it is not exposed to such an elevated temperature, the thermal polymerization of the vinyl monomer is not carried out, but moreover, it may dissolve in the carbon dioxide of supercritical or a subcritical state, and a vinyl monomer may fully be removed.

[0013] The low-molecular-weight matter removed by this invention approach means the oligomer which consists of units, such as an unreacted object of a functional-group content siloxane and dimethylsiloxane, and it to about ten pieces. Moreover, especially the molecular weight of the polysiloxane which constitutes the silicone oil set as the object of purification by this invention approach is usually abbreviation, although not limited. It is preferably applicable about the silicone oil which consists of a polysiloxane including the siloxane unit to about 400.

[0014] The critical points of a carbon dioxide are about 31.0 degrees C and about 75.3kg/cm², and it is referred to as being in a supercritical condition to be in the condition of having exceeded this and to have the in-between property of a liquid and a gas, i.e., the consistency near a liquid, and a diffusion coefficient near a gas. Moreover, although there is no clear definition, generally a subcritical state exists near the supercritical condition, is about 50kg/cm² in the temperature of about 25 degrees C or more, and pressure in a carbon dioxide, is in the above-mentioned critical temperature or below a pressure, and says what has the in-between property of the above liquids and a gas.

[0015] When the conditions used for the usual supercritical extraction and a subcritical extract can be used in operation of this invention approach and 80 - 500 kg/cm², 50 to 120 degree C, and a subcritical extract are usually included in the case of supercritical extraction, it is desirable to carry out using the carbon dioxide in a supercritical condition or a subcritical state under 70 - 500 kg/cm² and conditions of about 25-120 degrees C. The equipment used for the usual supercritical extraction and a subcritical extract can be used for the equipment for enforcing this invention approach. this invention approach can be performed to drawing 1 using the equipment shown roughly. In drawing 1, a carbon dioxide is supplied to a compressor 3 via a heat exchanger 2 from the carbon-dioxide cylinder 1, and it compresses to a predetermined pressure. The carbon dioxide compressed into the predetermined pressure is further made into extract temperature predetermined by the heat exchanger 4, and it is made supercritical or a subcritical state, and introduces to the extract tub 5. The extract tub 5 prepared by predetermined temperature is filled up with the conversion silicone oil which should be refined, the carbon dioxide containing the low-molecular-weight matter after extracting is decompressed through a reducing valve 6, it introduces into the separation tub 7, and the low-molecular-weight matter is separated. The carbon dioxide which separated the low-molecular-weight matter is recycled via a heat exchanger 2.

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TECHNICAL PROBLEM

[The technical problem which should solve invention] Therefore, the object of this invention is offering the approach supercritical extraction's being able to extract the low-molecular-weight matter from the denaturation silicone oil which consists of a polysiloxane containing a functional group efficiently, and denaturation silicone oil's being refined.

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